PATENT SPECIFICATION

NO DRAWINGS

1,159,445

1,159,445

Date of Application and filing Complete Specification: 12 June, 1967.
No. 27019/67.
Application made in France (No. 65210) on 13 June, 1966.
Complete Specification Published: 23 July, 1969.

Index at acceptance: —C1 B(3C1, 3C3, 3C4, 3C5, 3CX, 4); B5 A(1L, 2A1, 2A4B)
Int. Cl.: —C 05 b 19/00

COMPLETE SPECIFICATION

Method of Granulating Fertilisers

We, PRODUITS CHIMIQUES PECHINEY-SAINT-GOBAIN, a Body Corporate organised under the laws of the French Republic, of 16 Avenue Matignon, Paris 8e, France, do hereby declare the invention for which we pray that a patent may be granted to us and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to a method of granulating fertilisers.

In the manufacture of fertilisers in granular form, a wet fertiliser mass which has to be partially dried can be used, or else fertiliser salts in powder form can be treated with water or aqueous solutions, so that the granular end product contains the amount of water generally acknowledged to be necessary, namely about 15% related to the raw materials. However, the drying of the granules thus formed and the removal of the water vapour that is to be eliminated cannot be done without deterioration due to bursting of the granules or to loss of ammonia. When the product is subsequently screened, much of the fertiliser is of unsuitable grain size and has to be recycled through the granulating process. After the process has been started up, therefore, part of the material fed to the granulating process consists of recycled product and the rest is fresh material. The ratio of the weight of recycled product to the weight of fresh material during a given period of time, is referred to in this specification as the "recycling rate". In the known processes, this recycling rate is generally higher than 4. Moreover, the granules obtained are porous to some extent and do not remain in proper condition in the presence of moisture, so that they often need protection by being coated or dusted with inert substances.

The method according to the invention, which can be applied continuously or as a batch process, makes it possible to produce granular fertilisers which are regular in shape and size, dry, hard and compact, with a recycling rate, as defined above, of the order of 1 and generally lying between 0.7 and 1.5.

According to the invention, a method of dry granulating fertilisers comprises subjecting substantially dry fertiliser ingredients in powder form to simultaneous agitation and heating in such a way and at such a temperature that superficial softening occurs. Optionally, the fertiliser ingredients may be passed through a preliminary stage, in which they are mixed and dried until a dry powder is obtained, before being subjected to the actual granulating treatment.

At least one of the fertiliser ingredients may be a phosphate, nitrate, chloride or sulphate of potassium, ammonium or magnesium. One of the ingredients is preferably a salt containing nitrogen in nitrate and/or ammonium form. Ingredients may be mixed in proportions such as to produce a mixture having a desired N—P—K formula.

The fertiliser ingredients are preferably solid powders, with grain sizes lying generally from 0.2 mm. to 1.5 mm. They preferably have a moisture content of 0.2% or less.

Some of the fertiliser ingredients, during mixing or during any preliminary drying, may be in the form of a highly concentrated aqueous solution, for example one containing 95% to 99% of ammonium nitrate, alone or accompanied by ammonium phosphate and/or sulphate. The moisture content of such a mixture is preferably less than 1%.

Superphosphates with a moisture content of for example 5% to 10% may be used during this preliminary stage, as part ingredients of the fertilisers.

The method is carried out by agitating the

[Price

fertiliser ingredients so as to maintain intimate contact between its constituent particles and to bring those particles into repeated contact with the container walls, heat being applied at the same time, so that part of the constituents undergoes superficial softening.

If granulation is to be preceded by drying, the apparatus used preferably comprises two main units: one unit in which to carry out the mixing and drying constituting the preliminary stage of the method, for which purpose any conventional type of apparatus may be used; and a second unit in which to carry out the agitating and heating constituting the main part of the method, for which purpose use may be made of any conventional type of apparatus incorporating some means of internal heating, or external heating, such as a jacket. It is preferable for the heat to be applied by means of heated walls.

One particular advantageous form of apparatus for continuous working combines these two main units in one. Thus, the apparatus may consist of a tube rotating about its own axis, through which the fertiliser ingredients travel, being mixed and dried in the first part and then churned and heated in the second part. The mixing and drying are effected mainly by the rotation of the heated tube and the heat is applied preferably by the circulation of a hot fluid through a jacket.

After granulation, the resulting granular products can be subjected to partial cooling, for example to the extent of 5 to 10 Centigrade degrees, screening, re-cycling of the material of unsuitable grain size and then the final cooling of the marketable product. In general terms, the product to be regarded as of marketable quality will be of normal grain size, with granules of 2 to 4 mm. in diameter.

When the process is carried out continuously in a rotary tube, more than 50% of the product is of marketable grain size, and the granules obtained are substantially spherical.

The granule size can be varied by varying the working conditions, such as the temperature of the apparatus, the time the ingredents remain within it or, again, the size of the units.

The apparent density of the granular fertilisers produced is generally from 0.9 to 1.1 g./c.c., according to their composition and grain sizes.

The hardness is determined by measuring the load required to crush a granule of given size. To do this, tests are carried out on 10 granules of between 2 mm. and 2.5 mm. diameter. One such granule is placed under the movable plate of the test equipment and this plate is loaded with lead pellets, the load being progressively increased at the rate of 3 kg./minute until the granule breaks. Further weight measurements of this kind are then taken for each of the remaining 9 granules, and the average of the 10 measurements is taken.

The hardness of the product according to the invention, as determined by the method described above, is from 5 kg. to 10 kg., whereas that of fertiliser granules made by existing wet-granulation processes is generally from 2 kg. to 5 kg.

The free moisture content of the granules of this product does not exceed 0.5% by weight, whatever raw materials be used, and is generally from 0.01% to 0.1% for formulae not containing superphosphates.

Agglomeration is measured as follows:

A sample of 50 g. of the product is placed in a cylindrical press 50 mm. in diameter, loaded with a weight equivalent to 12 tons/sq.m. The sample is left in for 12 days at a temperature of 18°C. to 20°C. This gives a "cake", the breaking strength of which is measured by placing it under a cylinder 3 mm. in diameter, on which a regularly increasing pressure is exerted until the "cake" breaks.

The breaking strain expressed in kg. is termed the "agglomeration".

The agglomeration of the granular fertiliser according to the invention, found by the test thus defined, is mostly nil or very low and is always less than 3 kg., without the use of anti-agglomerants.

The surface of the granules is smooth in appearance and their porosity, measured by mercury porosimeter, is approximately 0.01 c.c./g. for fertiliser not treated with anti-agglomerants. Although it is not usually necessary, the agglomeration and porosity of the granules can be further reduced by anti-agglomeration treatment with, for example, inert substances such as clays.

The method according to the invention can be applied to a mixture of fertiliser salts containing a proportion of inert material or ballast or small quantities of substances capable of improving the agricultural properties of the fertilisers.

The method according to the invention has the advantage of making possible a re-cycling rate 4 to 7 times lower than is the case with most known methods. The size of the apparatus required is therefore reduced.

The power expended in likewise smaller, by virtue of the fact that only very little water has to be driven out in vapour form during drying. The air or gas circulation during granulation is greatly reduced, so that the risk of ammonia loss, which is appreciable particularly when compositions rich in nitrogenous ingredients are to be granulated, is lessened.

Contrary to compression - granulation

methods, the method according to the invention does not call for high pressures.

The finished product has a regular grain size distribution and the granules, because of their smooth surface and compactness, are excellent for storing.

By comparison with fertilisers granulated by the known methods in the presence of water, those granulated in accordance with the invention offer appreciable advantages in use, especially in view of their low hydroscopicity and the regular shape, which makes them flow freely.

The method can be applied to fertilisers of the most diverse composition.

The following six practical Examples are given to illustrate the carrying out of the method according to the invention. In these Examples, all quantities expressed as "parts" are parts by weight.

EXAMPLE 1

Into a unit consisting of a drying and granulating cylinder with heated walls, the first part for drying and the second part for granulation, were placed 319 parts of monoammonium phosphate (containing 36 parts of ammonia N and 172 parts of P_2O_5), 392 parts of ammonium nitrate (containing 68 parts of nitrate N and 68 parts of ammonia N) and 287 parts of potassium chloride (172 parts of K_2O). These are the ingredients for a fertiliser having an N—P—K formula of 17/17/17.

These were mixed and dried in the first part of the cyclinder, and then granulated in the second part. The wall temperature was regulated so that the granulation temperature in the second part was between 123 and 127°C.

Of the granules emerging, 50% to 60% were of diameters of 2 to 4 mm, the recycling rate therefore being 1½. Their hardness was 6 to 10 kg.

Their moisture content was less than 0.1%. Their agglomeration, in the absence of an anti-agglomerant, was 0.5 to 2 kg.; it was nil when an anti-agglomerant was used.

EXAMPLE 2

Into a unit consisting of a drying and granulating cylinder equipped with internal flame heating, the first part for drying and the second part for granulating, were placed 319 parts of monoammonium phosphates (containing 36 parts of ammonia N and 172 parts of P_2O_5), 392 parts of ammonium nitrate (containing 68 parts of ammonia N and 68 parts of nitrate N) and 287 parts of potassium chloride (172 parts of K_2O). These are the ingredients for a fertiliser having an N—P—K formula of 17/17/17.

These were mixed and dried in the first part, and then granulated in the second part at a temperature of 123°C. to 127°C.

Of the granules emerging, 50% to 60% were between 2 mm. and 4 mm. in diameter, the recycling rate therefore being 1½.

Their hardness was 6 kg. to 10 kg.

Their content of free moisture was less than 0.1%.

Their agglomeration, in the absence of anti-agglomerant, was 0.5 kg. to 2 kg.; it was nil when an anti-agglomerant was used.

EXAMPLE 3

The same procedure as in Example 2 was followed with 424 parts of monoammonium phosphate (containing 48 parts of ammonia N), 106 parts of superphosphate 45 (containing 48 parts of P_2O_5), 260 parts of ammonium nitrate (containing 44 parts of ammonia N and 44 parts of nitric N) and 190 parts of potassium chloride (containing 115 parts of K_2O)—the ingredients for a fertiliser having an N—P—K formula of 13.5/27.5/11.5.

These ingredients were mixed and dried in the first part of the apparatus and then granulated at a temperature of between 115 and 120°C. in the second part.

Of the granules emerging, 50 to 60% were spherical and 2 to 4 mm. in diameter, had a hardness of between 5 and 7 kg., contained from 0.10 to 0.30% of free moisture and had an agglomeration of 1 to 2.5 kg. without anti-agglomerants and nil when an anti-agglomerant was used. The recycling rate is therefore 1½.

EXAMPLE 4

The same procedure as in Examples 2 and 3 was followed with 195 parts of monoammonium phosphate (containing 22 parts of ammonia N and 105 parts of P_2O_5), 100 parts of neutralised superphosphate 45 (containing 46 parts of P_2O_5), and 300 parts of potassium chloride (containing 180 parts of K_2O and 123 parts of ballast)—the ingredients for a fertiliser having an N—P—K formula of 12/15/18.

These were dried and mixed in the first part of the apparatus, and then granulated at a temperature of between 115 and 120°C. in the second part.

In the resultant product, 50% of the granules were between 2 and 4 mm. in diameter, the recycling rate therefore being 1.

Their hardness was 5 to 6 kg. and their free moisture amounted to 0.4 to 0.5%. The agglomeration was 1 to 2.5 kg. without anti-agglomerant and nil when an anti-agglomerant was used.

EXAMPLE 5

Into a unit consisting of a rotary cylinder with heated walls, the first part for drying and the second part for granulation, was placed a mixture composed of ingredients corresponding to an N—P formula of 25/25, containing 570 parts of ammonium nitrate

and 410 parts of monoammonium phosphate, with a moisture content of 10 parts, and recycle material, having the N—P formula 25/25. The wall temperature was regulated so as to keep the granulation temperature in the second part between 135 and 140°C. Of the granules emerging, 50% were between 2 and 4 mm. in diameter, the recycling rate therefore being 1. Their physical properties (hardness, moisture content and agglomeration) were of the same order as in Example 1.

EXAMPLE 6

The same procedure as in Example 5 was followed with 700 parts of the same mixture of ammonium nitrate and monoammonium phosphate to 292 parts of potassium chloride and recycle material. The wall temperature was regulated so as to keep the granulation temperature in the second part between 120 and 125°C. Of the granules emerging, 50% to 60% were between 2 and 4 mm. in diameter, the recycling rate therefore being 1⅓. Their composition corresponded to an N—P—K formula of 17.5/17.5/17.5 and their properties were similar to those of the granules described in Example 1.

WHAT WE CLAIM IS:—

1. A method of dry granulating fertilisers which comprises subjecting substantially dry fertiliser ingredients in powder form to simultaneous agitation and heating in such a way and at such a temperature that superficial softening occurs.

2. A method according to claim 1, wherein at least one of the fertiliser ingredients is a phosphate, nitrate, chloride or sulphate of potassium, ammomium or magnesium.

3. A method according to claim 1 or 2, wherein at least one of the fertiliser ingredients is a nitrate or an ammonium salt.

4. A method according to anly one of the preceding claims, wherein the fertiliser ingredients also include a superphosphate.

5. A method according to any one of the preceding claims, wherein the fertiliser ingredients are solid powders with grain sizes of 0.2 mm. to 1.5 mm.

6. A method according to any one of the preceding claims, wherein the fertiliser ingredients have a moisture content of 0.2% or less.

7. A method according to any one of the preceding claims, wherein the substantially dry fertiliser ingredients in powder form are obtained by subjecting moisture-containing fertiliser ingredients to a preliminary stage in which they are mixed and dried until a substantially dry powder is obtained.

8. A method according to claim 7, wherein the moisture-containing fertiliser ingredients subjected to the preliminary stage of mixing and drying contain one or more ingredients in the form of highly concentrated aqueous solutions.

9. A method according to claim 8, wherein said highly concentrated aqueous solution contains 95 to 99% by weight of ammonium nitrate, along or accompanied by ammonium phosphate and/or sulphate.

10. A method according to claim 9, wherein the moisture content of the moisture-containing fertiliser ingredients subjected to the preliminary stage is less than 1%.

11. A method according to any one of claims 7 to 10, wherein the moisture-containing fertiliser ingredients subjected to the preliminary stage include superphosphates having a moisture content of 5 to 10%.

12. A method according to any one of claims 7 to 11, wherein the preliminary stage of mixing and drying of the fertiliser ingredients and the agitating and heating to cause superficial softening take place in separate apparatus.

13. A method according to any one of claims 7 to 11, wherein the preliminary stage of mixing and drying of the fertiliser ingredients and the agitating and heating to cause superficial softening take place in the same apparatus.

14. A method according to any one of the preceding claims, wherein the simultaneous agitating and heating to cause superficial softening are carried out in apparatus provided with internal heating.

15. A method according to any one of the preceding claims, wherein the simultaneous agitating and heating to cause superficial softening are carried out in apparatus with heated walls.

16. A method according to any one of the preceding claims, carried out in one or more cylinders rotating about a substantially horizontal axis.

17. A method of granulating fertilisers according to claim 1 substantially as herein described.

18. Granular fertilisers when obtained by the method claimed in any one of the preceding claims.

19. Granular fertilisers according to claim 18, having granules of substantially sperical shape.

20. Granular fertilisers according to claim 19, the granules of which have a diameter of 2 to 4 mm.

21. Granular fertilisers according to any one of claims 18 to 20, having an apparent density of 0.9 to 1.1 g./c.c.

22. Granular fertilisers according to any one of claims 18 to 21, having a hardness, as herein defined, of 5 to 10 kg.

23. Granular fertilisers according to any one of claims 18 to 22, having a free moisture content of less than 0.5%.

24. Granular fertilisers according to any one of claims 18 to 22, containing no super-

osphates and having a free moisture content of less than 0.1%.

25. Granular fertilisers according to any one of claims 18 to 24, having an agglomeration, expressed as hereindefined as the breaking strain of a sample previously agglomerated in a press, of less than 3 kg. in the case of granules not treated with anti-agglomerants.

26. Granular fertilisers according to any one of claims 18 to 25, having a porosity of 0.01 c.c./g. in the case of granules not treated with anti-agglomerants.

For the Applicants,
RAWORTH, MOSS & COOK,
Chartered Patent Agents,
38 Sydenham Road,
Croydon,
Surrey, CRO 2EF.
— and —
75 Victoria Street,
London, S.W.1.

Printed for Her Majesty's Stationery Office by the Courier Press, Leamington Spa, 1969.
Published by the Patent Office, 25, Southampton Buildings, London, W.C.2. from which copies may be obtained.